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## Slow Release of Surfactant Utilizing Silica Nanosized Capsules

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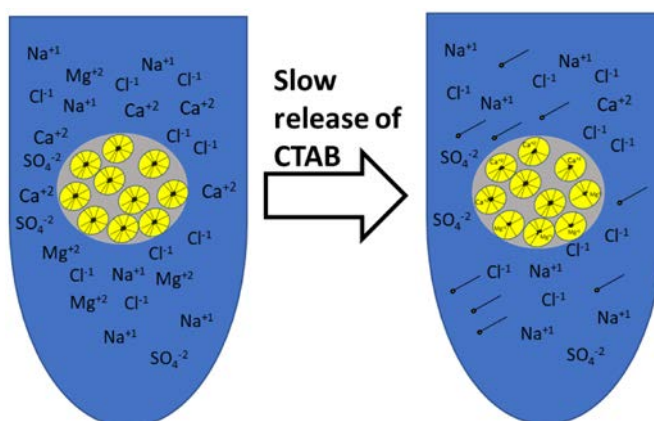
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### ABSTRACT

Engineering and scaling-up nanocarriers for the controlled release of surfactant are imperative for enhanced oil recovery (EOR). Herein, the use of silica-based nano-sized porous capsules to slowly release surfactant in saline water is reported. The results indicate that the silica shell ensures the stability of the protected surfactants in the cores under harsh conditions. Almost negligible release was noticed at salt-free brine (DI water). In saline brine, the particles slowly released surfactants molecules. 46% of the total surfactant encapsulated was released after 12 days as quantified by the remaining organic content after mixing with brine. Scanning Electron Microscopy (SEM) analysis confirms the stability of the surfactant incorporated particles in saline water that contains 56000 mg/L of salts.



### INTRODUCTION

Crude oil is by far the most utilized energy resource around the world. It accounts for up to 35% of the energy consumed in 2017 according to British Petroleum (BP, 2018). There are 3 stages for recovering oil. Primary relies mainly on the reservoir energy to produce oil. In the secondary stage, because of the decline of reservoir pressure, water and/or gas is usually injected to retain the pressure and increase the production of

hydrocarbon. Tertiary recovery accounts for using either thermal or nonthermal methods. Thermal methods have been implemented since 1950. Thermal methods are best used for heavy oil fields (Thomas, 2008). The nonthermal methods include utilizing gas and chemicals either before or post seawater flooding. Example of gas flooding includes CO<sub>2</sub> combined with water in a Water Alternating Gas (WAG) mode (Kokal, Sanni, & Alhashboul, 2016). Another means of nonthermal flooding include injection of chemicals such as surfactants to increase the capillary number and/or improve the mobility ratio. The capillary number is a function of viscosity, velocity of displacing fluid and interfacial tension and is given by  $N_c = \frac{\mu v}{\sigma}$ . One of the ways to increase the capillary number, and hence displace the residual oil, is by decreasing the interfacial tension, accomplished typically by utilizing surfactants (Thomas, 2008).

In recent years, there have been intensive efforts to find chemicals that could enhance oil recovery beyond the saturation limit. This requires agents to be able to change the forces that hinder the oil in the reservoir. For example, it has been studied and reported that after a long time of production, the reservoir wettability changes from favorable water-wet, mixed-wet region to oil-wet (Peng, et al., 2017). Hence, in order to recover the oil, the chemicals need to change the wettability from oil-wet to water-wet.

Several publications reported that surfactants enhance oil recovery. Moreover, others demonstrated that the use of polymers and surfactant improved the mobility ratio and increased oil recovery (Al-Shakry, 2018; Fletcher, Cobos, Jaska, Forsyth, & Crabtree, 2012; Iglauer, Wu, Shuler, Tang, & Goddard, 2010; Liu, Dong, Ma, & Tu, 2007). However, the use of bare surfactants has several drawbacks such as high rock adsorption, which will impose economic challenges, and instability at harsh reservoir conditions such as high temperature and concentration of electrolytes. Overcoming these challenges usually is accomplished by injecting a large amount of surfactant to enhance oil recovery. To overcome this issue, a possible solution is to encapsulate and controllably deliver surfactants for better EOR performance.

Incorporating NPs into petroleum reservoir has been recently heavily perused to give rise to a significant increase in oil recovery. A-Dots nanoparticles of size in the order of 5-10 nm were field tested in harsh environment of temperatures up to 100 °C and salinity up to 120,000 PPM and demonstrated extraordinary stability (Kanj M., Rashid, & Giannelis, 2011). Furthermore, Alaskar and Kosynkin (2016) reported successful field deployment of the A-Dots, that were able to travel 500 m in the harsh environment. Moreover, several authors have conducted experiments that utilized organic and inorganic nanoparticles and demonstrated their efficacy in wettability alteration and reducing the interfacial tension. For example, Amr et al (2017) have demonstrated a new class of nanosurfactants with sizes of 10-60 nm that achieved 7% extra oil recovery from the oil initially in place.

There are only few studies exploring the controlled slow-release in the oil industry. Nourafkan et al (2018) reported reducing the surfactant adsorption by 50% when using nanocarriers for Sodium Dodecyl Sulfonate (SDS) and achieved enhancement of 8% of oil recovery. However, previous studies did not address the kinetics of slow release of surfactant from porous materials to concentrated brine solution that simulate injected water. Moreover, no clear quantification of the amount of surfactant released is reported. The slow release of surfactant for oil recovery application is attractive since it can potentially minimize the loss of surfactant due to adsorption and provide better stability of the surfactant in the harsh reservoir conditions. Moreover, the slow release of a surfactant ensures maintaining the activity of the material as it travels in the reservoir for longer distances.

In this work, a scalable and facile fabrication of nanosized capsules for controlled slow-release of surfactants is described and its applicability to oil field application is demonstrated. The system is based on the self-assembly of surfactants in an inorganic material. The material resulted from this synthesis route was first proposed by Beck et al (1992). Its unique structure and simple synthesis make it attractive for several research areas. MCM-41 (Mobil Composition of Matter No.41) has been widely studied as a template for drug delivery (Vallet-Regi, Rámila, Del Real, & Pérez-Pariente, 2001; Muñoz, Rámila, Pérez-Pariente, Díaz, & Vallet-Regí, 2003; Datt, El-Maazawi, & Larsen, 2012) and as template for catalysts (Blasco, Corma,

Navarro, & Perez Pairente, 1995; Chen, Li, Pan, Ma, & Li, 2016). The utilization of MCM-41 in drug delivery and catalysis usually start by eliminating the surfactant from the template and then loading the required drug into the template. In contrast, in our system we utilize for the first time the readily available surfactant inside the mesopores and propose a controlled release mechanism for it at oil reservoir conditions.

As such this is the first report on using MCM-41 as silica nano-carrier to control the release of surfactant enhance the oil recovery. The MCM-41 showed resistant to the highly saline water mainly due to the silica shell stability. Almost negligible release was noticed at salts-free brine. In saline brine, the ions trigger the controlled slow-release of surfactant molecules. To that end, MCM-41 is most-likely best suited for ions-responsive nano-carrier to slowly release surfactant for EOR. The fact that the ions are readily available in the reservoir makes this approach more compelling. Moreover, we demonstrate how the nano silica capsules maintain their size even when being mixed with high salinity water.

## EXPERIMENTAL SECTION

### MATERIALS

*Reagents:* Cetyltrimethylammonium Bromide (CTAB), Tetraethylorthosilicate (TEOS), Sodium Hydroxide (NaOH), Sodium Chloride (NaCl), Calcium Chloride Dihydrate ( $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ), Magnesium Chloride Hexahydrate ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ), Sodium Sulfate ( $\text{Na}_2\text{SO}_4$ ) and Sodium Bicarbonate ( $\text{NaHCO}_3$ ) were purchased from Sigma Aldrich and used as received without further purification.

### SYNTHESIS

The brine formulation is shown in Table 1 and was similar to that proposed in the literature (Mashat, Abdel-Fattah, Aramco, & Gizzatov, 2018). The salts were added to 1 L of 18.2 MQ water and stirred at room temperature until the mixture became homogenous. The synthetic seawater was used as is without further purification.

Table 1—the amount of the salt used in the preparation of the salinity water used in the release experiments

Compound	NaCl	$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	$\text{Na}_2\text{SO}_4$	$\text{NaHCO}_3$
Weight (g)	41.04	2.384	17.645	6.343	0.165

To synthesize the porous nano-sized capsules containing CTAB, we followed the procedure of synthesizing Mesoporous Silica Nanoparticles as described in the literature (Ahmed & Ramli, 2011). Briefly the CTAB was dissolved in 480 ml of 18.2 MQ water. The mixture was stirred until all the CTAB dissolved. 2 M NaOH solution was prepared in 18.2 MQ water and 3.5 ml was added to the beaker containing CTAB and water followed by adjusting the temperature to 80 °C. After that, 5 ml of TEOS was added dropwise while maintaining vigorous stirring of the solution. The mixture was kept stirring for 2 hours. The resultant white precipitate was collected, washed several times with deionized water and centrifuged at 9000 RPM for 15 minutes. The powder was then dried at 50 °C for two days.

### METHODS

Thermogravimetric analysis (TGA) was conducted utilizing TA instrument Model Q500 under atmospheric conditions and a flow rate of 10 ml/min. The heating rate was at 10 °C/min. X-ray diffraction analysis was performed on a Bruker D8 Advance ECO Powder Diffractometer. The Scanning Electron Microscopy images were obtained using Tescan Mira3 FESEM. Transition Electron Microscopy (TEM) images were obtained utilizing Tecani 12 Spirit TEM STEM. Zeta Potential Measurements and Dynamic Light Scattering Measurements were carried out utilizing Zetasizer Nano ZS90 (Malvern Instrument) to determine the surface charge and the size of the particles.

Release experiments were conducted by dissolving 10 mg of the encapsulated surfactant in 20 ml of seawater. The solution was kept stirring to simulate the dynamic conditions of the reservoir, where fluids are in constant movement. Also, it is essential to avoid build-up of concentration gradient on the nanoparticles. After that, the sample was centrifuged at 8500 RPM for 15 minutes. The powder was placed in the oven at 50 °C to dry. The dried powder was then analyzed utilizing TGA to determine the amount of surfactant remaining in the capsules indicated by  $M(t)$ , which is the amount of surfactant released at time  $t$ . This amount is compared to the initial amount of encapsulated surfactant  $M_o$  and the released percentage is calculated as follow:

$$\text{Released CTAB Surfactant (\%)} = \frac{M_o - M(t)}{M_o} \times 100 \quad (1)$$

## RESULTS AND DISCUSSION

### Characterization

The initial amount of surfactant in the nanocapsules as measured by TGA is 40% (Figure 1). The weight loss from 130°C to 330°C indicates the total organic content present in the particles. The X-ray Diffraction shown in Figure 2 clearly shows the ordered porosity of the material with the surfactant in the porous spaces. The four peaks that are present at  $2\theta$  of 2.2°, 3.8°, 4.4° and 5.8° clearly indicate the long-range ordered mesoporosity of the particles. Moreover, TEM image in Figure 3 of the powder clearly shows the porous structure. The SEM image shown in Figure 4 indicates the presence of hexagonal particles with a practically uniform size of 50-100 nm in diameter.

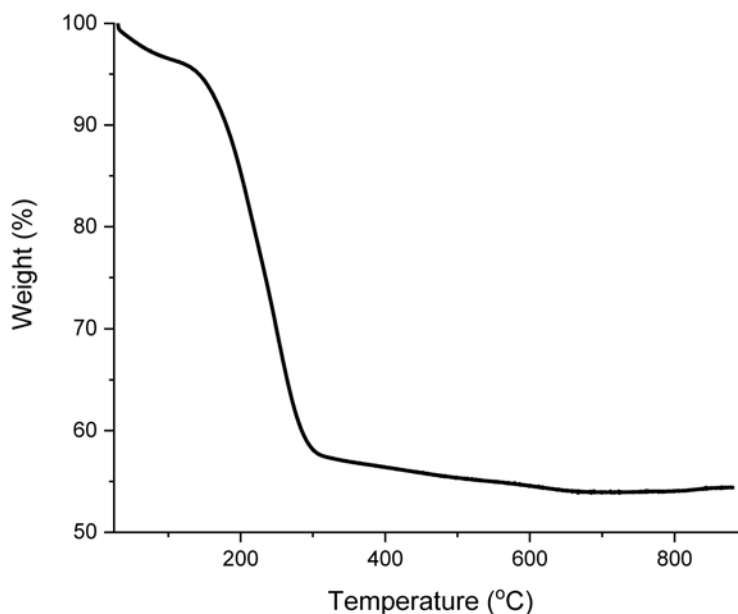


Figure 1—TGA results for the encapsulated surfactant indicates 40% of total organic content

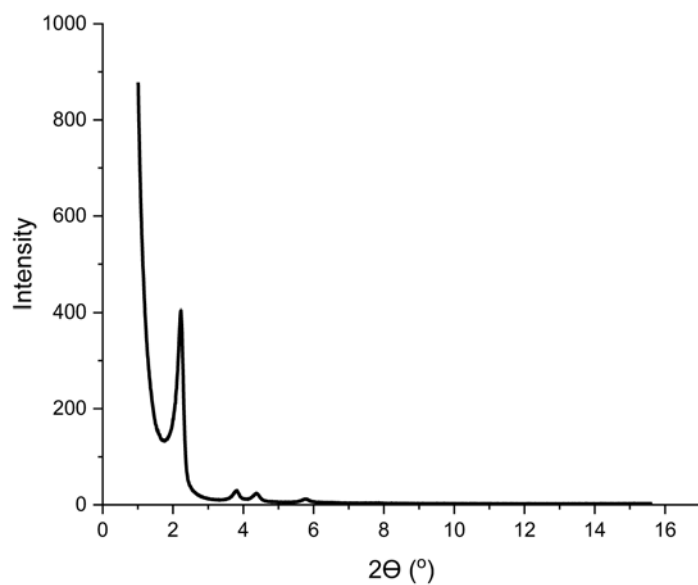


Figure 2—X-ray Diffraction pattern of MCM nanoparticles

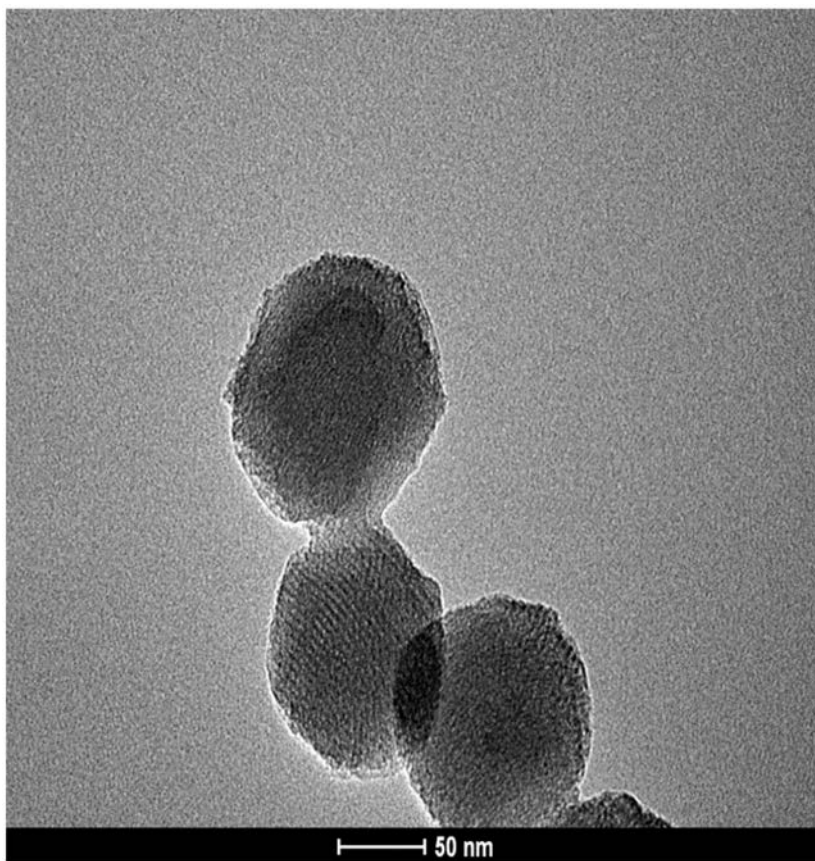


Figure 3—TEM image of the encapsulated surfactant showing the channels of the porous material



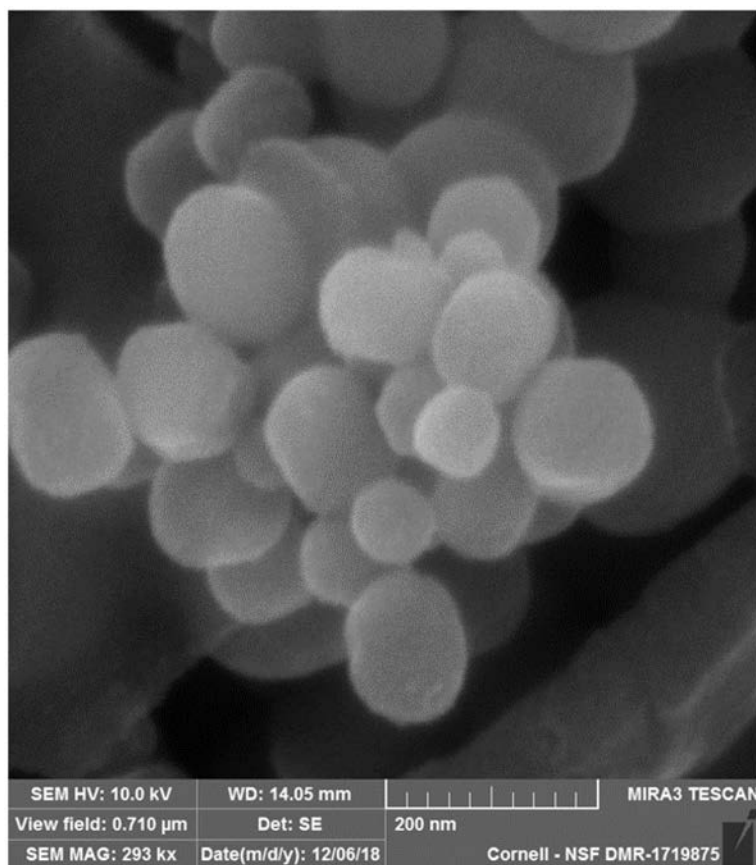


Figure 4—SEM images showing particles with hexagonal shape of size 50-100 nm

### Release Experiments

As indicated earlier, the release experiments were conducted by mixing 10 mg of the encapsulated surfactant in 20 ml of brine. The sample was removed from the suspension and allowed to dry at 50 °C. Finally the amount of organic content was determined by TGA.

Figure 5 shows the amount of CTAB released in 12 days. The release starts very slowly at 10% within the first day. It then continues to increase gradually until it stabilizes at about 50% within the first 12 days of experiments. Despite the burst release that occurred within the first hours, the particles maintain a slow release profile for two weeks ensuring controlled and sustained delivery mechanism of the surfactant. This unique behavior of the slow release of CTAB over the course of 12 days supports the idea of the potential use of this material as a carrier for surfactant that can be delivered efficiently in the reservoir without being lost quickly due to adsorption or instability. The release of surfactant, however, could be even further controlled with the introduction of functional groups and modification of physical properties of the particles such as the pore volume and pore size. Such techniques have been investigated extensively in drug delivery, where it was shown that the release could be controlled by the introduction of different surface groups (Manzano, et al., 2008) and slower release was achieved by reduction of the pore size (Wang, 2009).

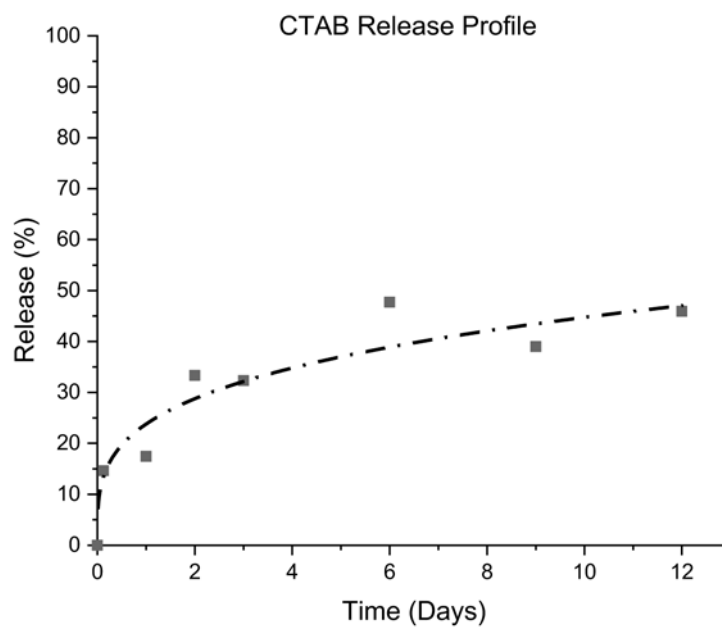


Figure 5—Release profile of CTAB over 12 days

Moreover, with minimum stirring speed, the particles maintained their dispersibility with the high salinity brine that is mixed with. As shown in Figure 6, the sample shows no apparent phase separation when mixed with high salinity water at both room temperature and 65 °C.

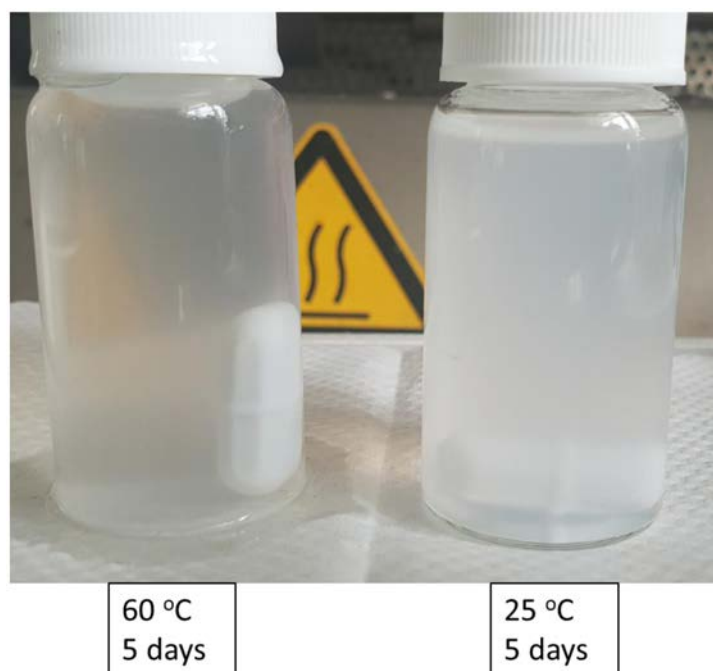


Figure 6—The dispersibility of the encapsulated surfactant with high salinity brine at both high temperature at room temperature

One limitation that some nanoparticles systems have shown upon mixing with divalent salts, is particle agglomeration. A phenomenon that is not desirable when using Nanoparticles to the challenging reservoir environment as they will plug the formation causing more decline in oil production. In constrast, our approach shows that the particles actually maintain their size by SEM and DLS as shown in Figures 7 and 8. One reason for this stability is the dominant negative charge at the surface of the particle. Previous studies

(Zhao, Lu, Whittaker, Millar, & Zhu, 1997) indicated that the predominant groups at MCM-41 particles are silanol groups. The zeta potential in Figure 9 indicates that the charge on the surface of the particle is about  $-25$  mv.

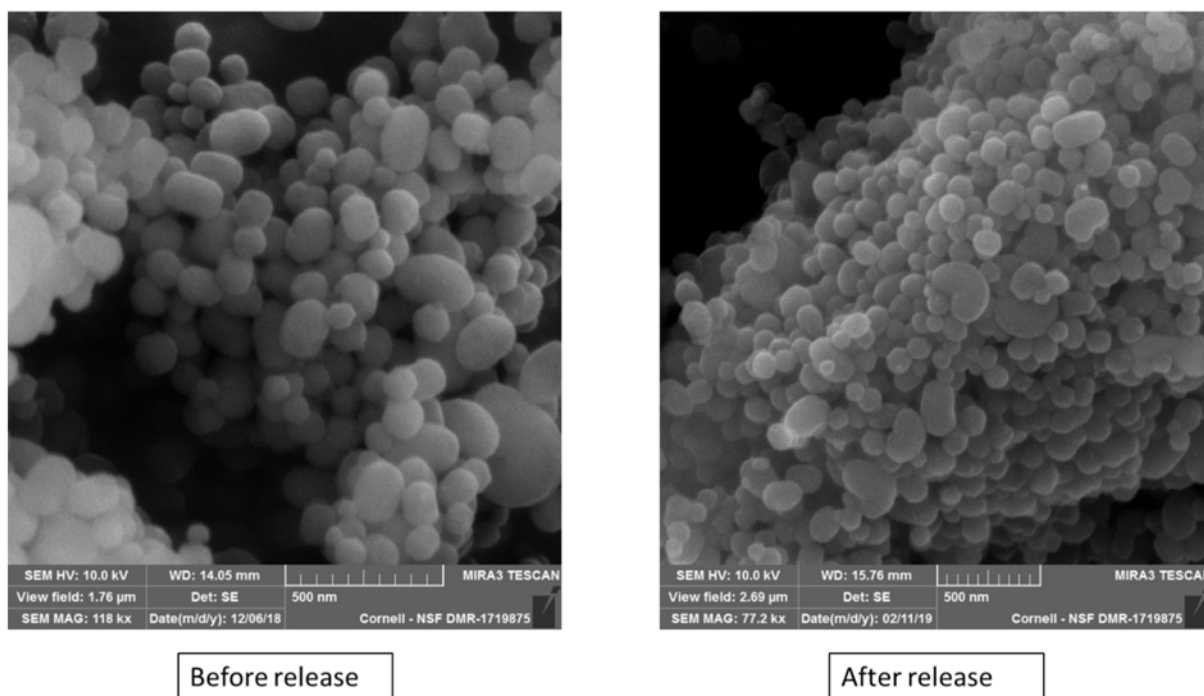


Figure 7—SEM images showing the capsules before and after the release of surfactant

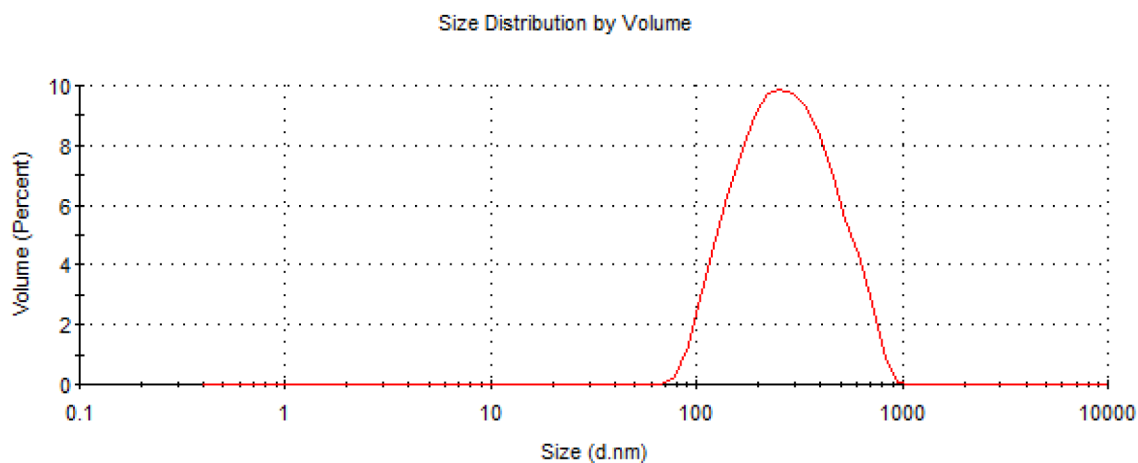


Figure 8—DLS show the particle maintain size of 200 nm upon mixing with high salinity brine solution



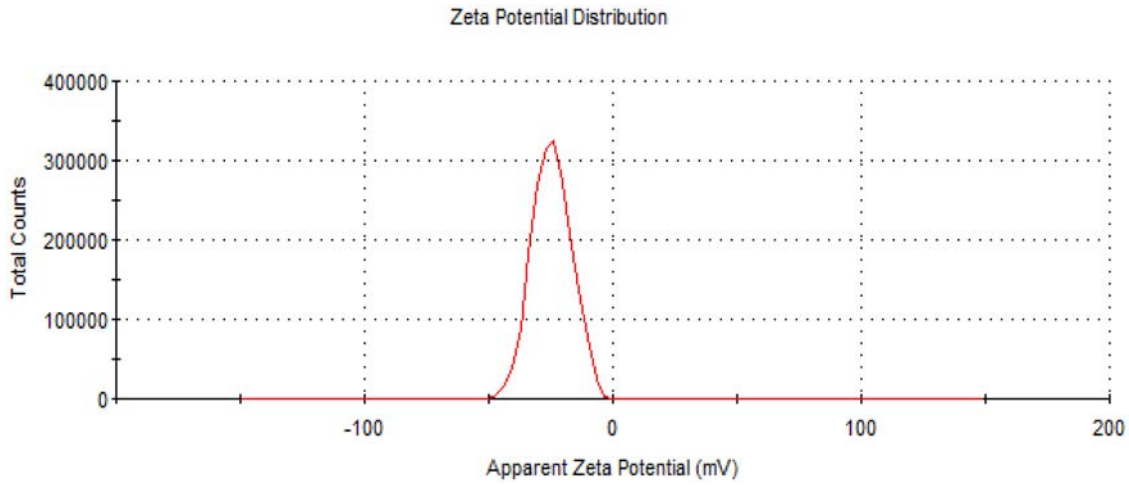


Figure 9—Zeta Potential of the nanosized porous particles indicate dominant negative charge

The SEM images after the release experiment indicate that the release does happen as a result of diffusion of surfactant molecules from the porous media to the bulk fluid. Moreover, it certainly indicates that the release does not occur because of the dissolution of the particles. In contrast, the surfactant migrates from the porous structure to the bulk fluid. Most importantly, this shows that the particles do maintain their size even when they are mixed with high salinity brine.

Within three days 35% of the surfactant originally encapsulated is released. The release profile might be considered as a burst release and it is certainly applicable whether the nano silica particles are injected in a producer then allowed to release around the well, or in an injector where they will travel in the reservoir. Asphaltene precipitation tends to occur near-wellbore area of producers where the pressure decline is noticeable. As a result of asphaltene deposition, wettability of the reservoir tends to be more oil-wet (Kim, Boudh-Hir, & Mansoori). It has been shown that CTAB is considered as one of the best wettability modifiers. Therefore, the slow release of CTAB from the Nanoporous particles around the wellbore of an oil-wet formation will result even in further reach in the reservoir.

The diffusion process of CTAB from the matrix could be further understood by considering this process to follow Fick's second law:

$$\frac{\partial C_A}{\partial t} = D_{AB} \frac{\partial^2 C_A}{\partial Z^2} + x_A (N_A + N_B) \quad (2)$$

$C_A$ : Concentration of CTAB ( $\frac{\text{mole}}{\text{ml}}$ )

$x_A$ : Mole fraction of CTAB

$N_A$ : Flux CTAB

$N_B$ : Flux of seawater

$D_{AB}$ : Diffusion Coefficient ( $\frac{\text{cm}^2}{\text{min}}$ )

Here, we assume that the mole fraction of CTAB is very low compared to that of water and hence no contribution of convective flux to the continuity equation. Moreover, because our material remains the same (no swelling or dissolution) after the release as shown in Figure 7, the diffusion coefficient is constant (Datt, El-Maazawi, & Larsen, 2012). Several models were proposed to study drug diffusing from the MCM-41 matrix. One of the widely used models to study drug release from the porous medium is Higuchi model (Higuchi, 1963). The model assumes pseudo steady-state and that the amount of drug initially present is larger than the solubility of the drug in the system. The equation Higuchi employed is:

$$M(t) = \sqrt{D_{eff}(2C_o - C_s)C_s t} \quad (3)$$

Deff: Effective diffusion Coefficient ( $\frac{cm^2}{min}$ )

$C_o$ : Initial Concentration per unit volume ( $\frac{mg}{ml}$ )

$C_s$ : Solubility of the drug (encapsulated material) in the system  $\frac{mg}{ml}$

t: time (min)

And could be simplified to the following:

$$M(t) = k\sqrt{t} \quad (4)$$

Figure 10 shows the fit of the released surfactant versus the square root of time as suggested by the Higuchi model for a non-moving boundary condition. The experimental data follow clearly the model and the pre-factors of the  $t^{1/2}$  are a function of solubility constant of the CTAB in brine, diffusion coefficient and the area of the particle. The previously studied systems using this model showed deviations from the equation as time increases (Datt, El-Maazawi, & Larsen, 2012). However, our data indicate that the release profile follows the linearized Higuchi model up to 6 days and then deviates from it as a result of reaching the maximum release possible at the conditions of the experiment. The Higuchi model does not take into account the leveling of the release after a given time and it just describes the profile as a linear increase with time. To the best of our knowledge, this is the first proposal to utilize the Higuchi model, which has been thoroughly utilized in studying the diffusion of drugs, for the release of surfactant from porous nanomaterial at high ionic concentration. Since the model describes the data well, it could be further utilized to optimize the system for controlling the release of surfactant for oil field applications. For example, from the linear fit, one can obtain the diffusion coefficient for varying physical properties of the particles, and then design fit-for-purpose porous particles that could be deployed to control the release of surfactant for EOR applications.

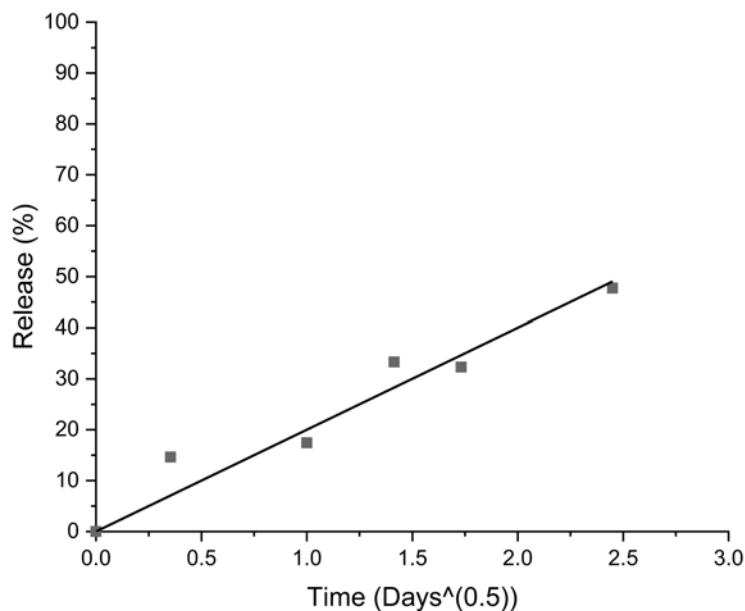


Figure 10—Model fit of the release data

The release of surfactant is driven by the readily available ions in brine. The ions that are abundant in an oil reservoir are the driving force for the release of surfactant molecules by reverse ion exchange. The nano-sized porous particles would only release surfactant upon encountering the ionic environment that is present in the brine and in the oil reservoir. To further investigate this theory, 10 mg of the material was mixed with 20 ml DI water and left stirring overnight. The sample was then centrifuged and dried at 50°C overnight. As shown in Figure 11, the weight loss between the as-synthesized Nanoparticles and the one aged for one

day are identical. When mixed with seawater, the nanosized particles released about 20% compared to none when mixed with DI water. This indicates that the release is due largely to the ions present in brine.

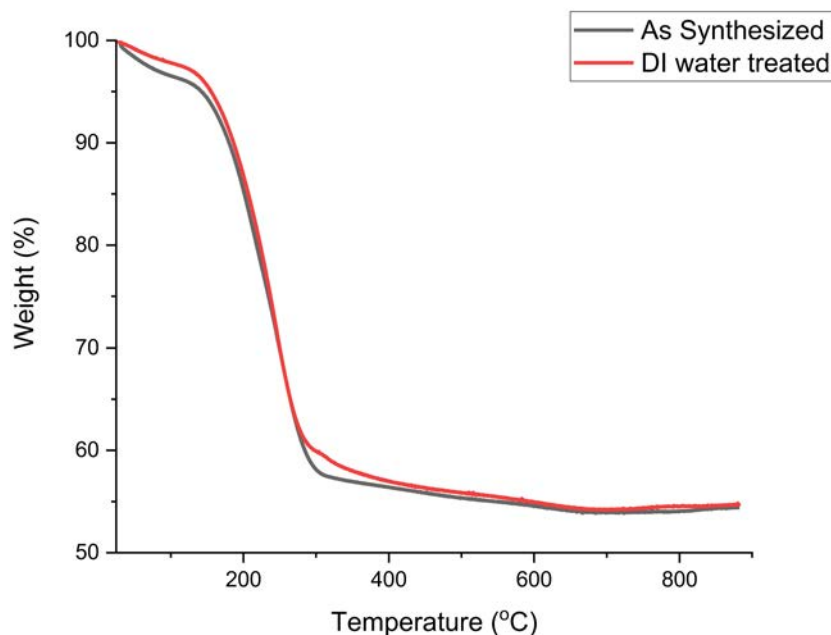


Figure 11—The weight loss comparing a sample aged with DI water for 1 day (red) and the sample before it is mixed with water (black)

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